EFFECT OF QPQ TREATMENT ON THE PERFORMANCE FOR H13 DIE STEEL

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Abstract

QPQ salt bath treatment of H13 steel was conducted by nitriding at the same temperature of 565 ℃ for various times, followed by the same post-oxidation process. Optical microscope, micro-hardness tester, X-ray diffraction and wear resistance tester were employed to characterize the microstructure, phase constituents, micro-hardness and wear resistance of the treated specimens. The results showed a compound layer mainly composed of ε-$Fe_{2-3}N$ and diffusion layer were formed during salt bath nitriding and a thin oxide layer composed of Fe_3O_4 was formed by post-oxidation, and the compound layer thickness increases with the nitriding time. The maximum surface hardness value of 1441 $HV_{0.3}$was obtained after nitriding at 565℃ for 150 min, which is as three times high as that of untreated sample. Meanwhile the wear resistance of H13 steel is significantly improved by QPQ treatment, 150 min is the optimum nitriding time to improve the surface hardness and wear resistance of H13 steel.

Keywords: QPQ treatment, H13 steel, Wear resistance, Hardness, Microstructure

1 Introduction

H13 steel is a kind of widely used hot die steel due to its good mechanical properties at elevated temperature and moderate cost [1-3]. In real application, hot working tools are generally repeatedly subjected to high temperature and heavy loads [4, 5], these severe conditions tend to result in failure of the tools due to wear or plastic deformation etc. [6-12]. In order to improve the related properties of H13 die steel, researchers have tried various surface modification methods, such as plasma nitriding, ion implantation, laser surface treatment and carbonitriding [13-17], among these technologies, plasma nitriding is currently widely adopted in real application to improve the wear resistance[18-20]. Unfortunately, it generally takes dozens of hours to obtain the required properties, which brings out low efficiency and high consumption of energy [21, 22].

It has been reported that QPQ treatment is an effective surface modification technology, which has been widely used in manufacturing field to improve the wear and corrosion resistance of the components [23, 24], since a compact oxide film can be formed on the top of a nitriding layer on the metal surface after QPQ treatment. More importantly, comparing with plasma nitriding, QPQ treatment has significant advantage of much higher efficiency; it generally takes only several hours for the whole process.

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However, few reports can be found for the effect of QPQ treatment on the performance of H13 steel. The goal of this study is to systematically investigate the effect of QPQ treatment on wear behaviour of H13 steel, and thus determine the optimum processing parameters from the viewpoint of wear resistance.

2 Experimental

The experimental material is H13 die steel and its chemical composition (wt. %) includes: Si: 0.80~1.20, C: 0.32~0.45, Mo: 1.10~1.75, Cr: 4.75~5.50, Mn: 0.20~0.50, V: 0.80~1.20 and balance Fe. The specimens with size of 10 mm×10 mm ×10 mm were prepared for optical microscopy and micro-hardness test, with the size of 5mm thick and 32mm diameter disc for wear test. The specimens were quenched at 930℃ and tempered at 580℃. The specimen surface was ground using different granularity SiC papers (240, 500, 1500, 2000mesh) to achieve a mirror finish. Finally, the specimens were cleaned by ultrasound in anhydrous ethanol to remove the grease on the surface.

Firstly, the specimens were pre-oxidized at 350℃ for 25min in air furnace. Then the specimens was put into the salt bath nitriding medium at 565℃, and held for a time range of 60min~180min. Finally, the samples was put into the salt bath oxidation medium at 430℃ and held for 40min.

The cross sectional microstructure was observed by optical metallography with a type of XUG-05, phase constituents were determined by XRD with a type of D_{max} 2500 using Cu–Kα radiation, hardness was measured by a HXD-1000TMC micro-hardness tester, with holding duration of 15s, each hardness value was obtained by averaging no less than 5 measurements. Wear resistance was tested by a MMW-1A Wear Test Machine under ambient condition (20±2 ℃ and 50%RH). During the test, a GCr15 steel ball with 6 mm diameter was rotated at a speed of 200rpm and a test load of 200N on the surface of the samples for 60 min. The weight of the samples before and after wear test was measured by a balance accurate to 0.1mg, and then the weight loss was obtained by calculating the difference of the two weights.

3 Results and discussions

3.1 Cross-sectional microstructure

Fig. 1 shows the cross-sectional microstructure of samples after QPQ treatment under different conditions. It clearly showed that the compound layer and diffusion layer was formed from the sample surface to the substrate after QPQ treatment under all conditions. The compound layer thickness increases with nitriding time. The compound layer can significantly improve the hardness and wear resistance of H13 steel. But when the nitriding time is 180min, a loose structure is formed on the compound layer (**Fig. 1** (e)), which brings out side effect on the performance of H13 hot working steel. In addition, between the compound layer and substrate, a diffusion layer with darker color can be distinguished from Fig. 1, which benefits to improve the fatigue strength of H13 hot working steel. It has to be noted that a thin oxide layer composed of Fe_3O_4 was actually formed on the outmost surface during QPQ treatment, though it can not be distinguished by optical microscopy, which can be confirmed by XRD, as illustrated in “3.2 XRD analysis”.

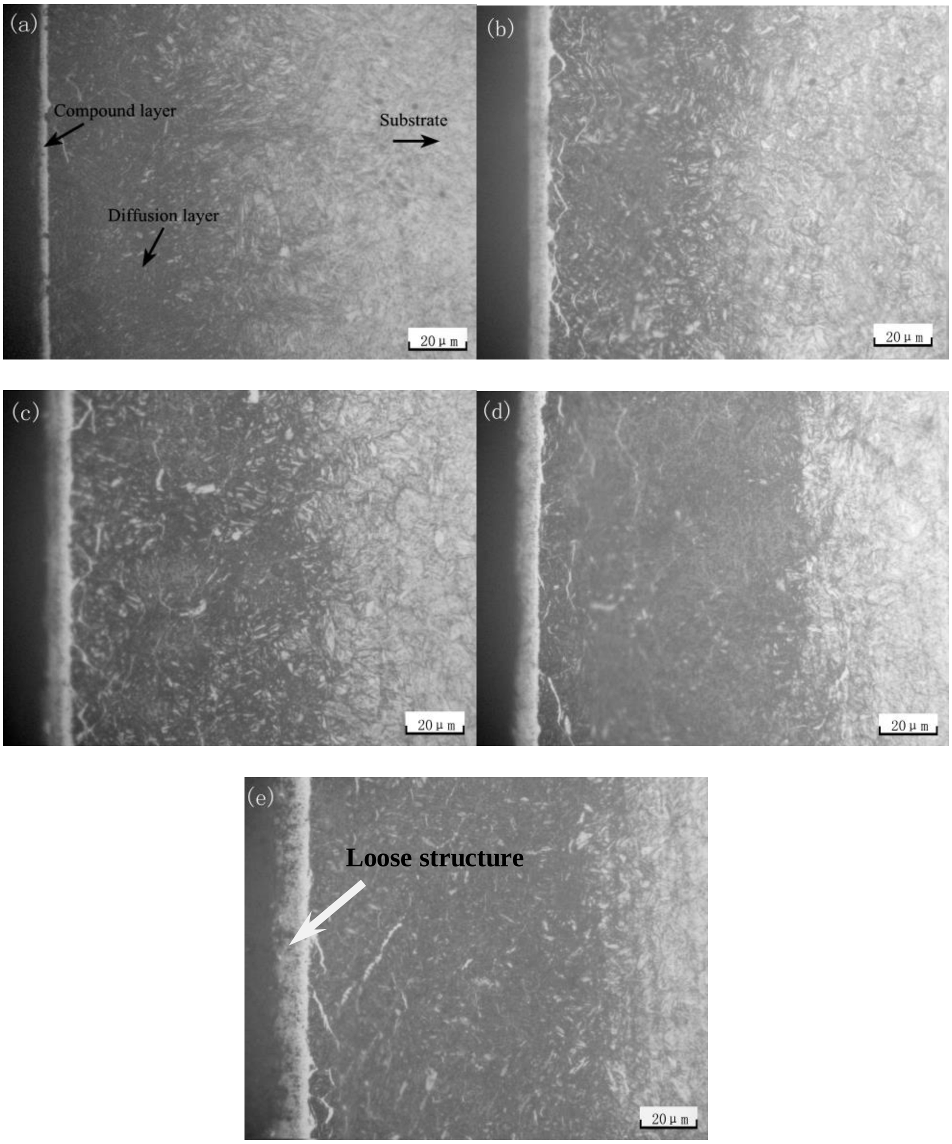

Fig. 1 The cross sectional microstructure of specimens nitrided at 565℃ for different time followed by post-oxidation at 430℃ for 40min: (a) 60min (b)90min (c)120min (d)150min (e)180min

3.2 XRD analysis

Fig. 2 presents the XRD patterns of the samples nitrided at 565℃for 150min with and without post-oxidation. It clearly showed that the main phase of the compound layer is $\varepsilon\text{-Fe}_{2\text{-}3}\text{N}$ treated under different conditions. Compared **Fig. 2** (a) and (b), the Fe_3O_4 phase appeared for the samples treated with post-oxidation. The Fe_3O_4 phase with chemical stability and low friction coefficients are benefits to further improve the wear resistance.

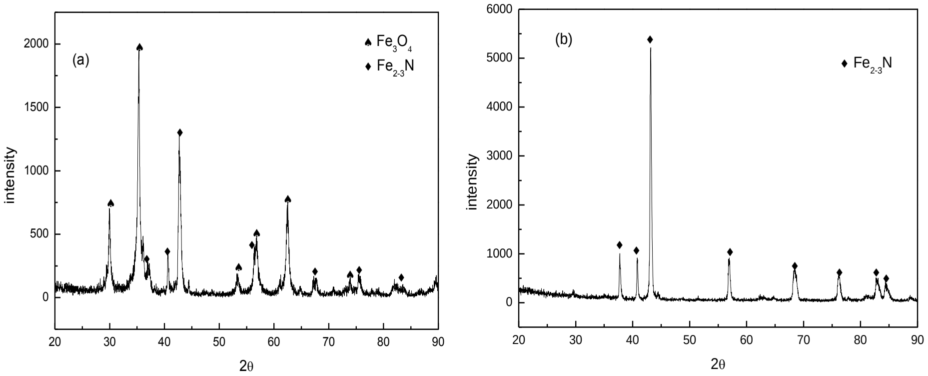

Fig. 2 XRD diffraction patterns of samples nitrided at 565℃for 150min post-oxidized at 430 ℃for 40min (b) without post-oxidation

3.3 Surface hardness

Fig. 3 presents the surface hardness of specimens nitrided for different time with the same post-oxidation. It can be seen that the surface hardness increases gradually with the nitriding duration and turns to decrease at 150min, with the maximum value of 1441HV$_{0.3}$. This is because the compact surface layer is getting thicker and thicker with nitriding time, until it turns to get loose at too long nitriding duration (**Fig. 1** (e)).

Fig. 3 also displays that the hardness value decreases as the load increase at the same nitriding time. That is because when the load is small, hardness indenter completely hits the nitrided layer. With the gradual increase of the load, the contribution of the matrix to the hardness value will increase. Therefore, the tested surface hardness value decreases as the testing load increases.

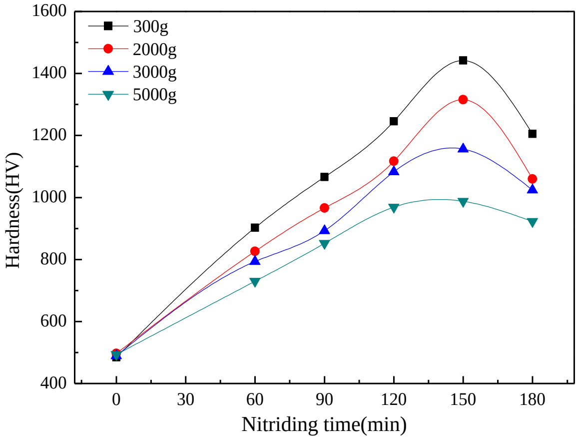

Fig. 3 The surface hardness profile of the specimens nitrided at 565℃for different time

3.4 Wear resistance

Fig. 4 shows the weight loss of samples untreated and treated by QPQ under different conditions. It can be seen that the weight loss of untreated sample is 0.0343g, which is much larger than that that treated by QPQ. The weight loss of QPQ treated samples is nitriding time dependent, and decreases gradually with the increase of nitriding time till 150min, then turns to increase, with the minimum value of 0.0131g at 150min.This is because the surface layer treated by QPQ is composed of compact nitride layer with high hardness and Fe_3O_4 with low friction coefficient. However, the weight loss increases at nitriding time of 180min, due to the loose structure formed at this long nitriding duration, leading to worse wear resistance. It is concluded that 150min is optimum nitriding time for H13 steel from the viewpoint of improving wear resistance.

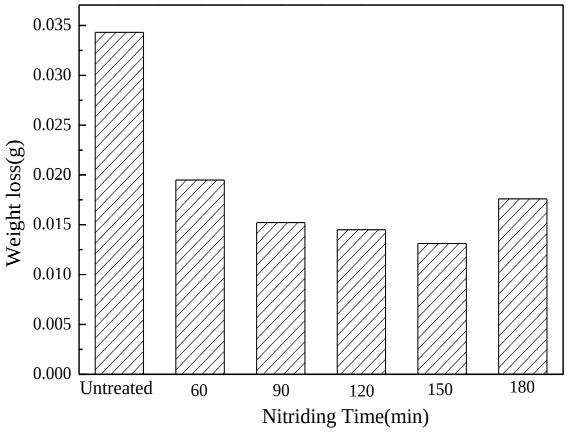

Fig. 4 The weight loss of samples untreated and nitrided at 565℃for different time

4 Conclusions

The QPQ treatment is adopted to improve the performance of H13 hot working steel. The results showed that diffusion zone and compound layer were found on H13 steel surface after QPQ treatment. The compound layer thickness increases with the nitriding time, and the compound layer kept uniform and dense when nitriding time was no more than 150min, but turned to be loose after that. The main phase of H13 steel treated by QPQ is $Fe_{2-3}N$ and Fe_3O_4, which can improve the performance of H13 steel. The surface hardness is significantly increased treated by QPQ, with the maximum hardness is of $1441HV_{0.3}$ at 565℃ for 150min, almost 3 times higher than that untreated. Meanwhile, the sample nitride at 565℃ for 150min has the best wear resistance. Therefore, 150min is the optimum nitriding time to improve the surface hardness and wear resistance of H13 steel.

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